PATENT ABSTRACTS OF JAPAN

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(54) MANUFACTURE OF TI-AL ALLOY SINTERED BODY

(57)Abstract:
PURPOSE: To manufacture a Ti-Al alloy sintered body excellent in high temp. strength and toughness by kneading Ti powder and Al powder applied with Ni plating having specified thickness with an organic binder, executing compacting and dewaxing, thereafter sintering it in an Ar atmosphere.
CONSTITUTION: The powdery material of Ti powder with Al powder applied with Ni plating having 0.05 to 1.0μm thickness is kneaded with an organic binder. As for the Ti powder and Al powder, the one having about ≤30μm average particle diameter is preferably used. Furthermore, the Ni plating can be executed by a electroless plating. As for the organic binder, the well-known one can be used, and as for the kneading, it is executed preferably by a kneader having high kneading power such as a pressure kneader. Then, the kneaded product is subjected to dewaxing in heating, and after the dewaxing treatment, sintering treatment is executed at 1200°C for about 2hr in the Ar atmosphere. In such a manner, the objective Ti-Al alloy sintered body excellent in high temp. strength and toughness is obtd. and by this method, the manufacture of lightweight heat resistant material parts having a complicated shape is enabled.

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TITLE: Titanium@-aluminium@ alloy sintered compact mfr. for aircraft engines - by mixing **nickel@-plated titanium@ powder** and aluminium@ powder, kneading with organic binder, compacting, degreasing and sintering

PATENT-ASSIGNEE: KAWASAKI STEEL CORP[KAWI]

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ABSTRACTED-PUB-NO: JP 05263165A

BASIC-ABSTRACT:

Ti-Al Alloy sintered compact is made by mixing **Ni-plated Ti-powder** and Al-powder, kneading with an organic binder, compacting the kneaded matter, degreasing and sintering.

USE - Used for aircraft engines. It has excellent high-temp. strength and toughness.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: TITANIUM@ ALUMINIUM@ ALLOY SINTER COMPACT MANUFACTURE AIRCRAFT
ENGINE MIX NICKEL@ PLATE TITANIUM@ POWDER ALUMINIUM@ POWDER KNEAD
ORGANIC BIND COMPACT DEGREASE SINTER

DERWENT-CLASS: M26

CPI-CODES: M22-H03B; M26-A02; M26-B06; M26-B06A;

CLAIMS

[Claim(s)]
[Claim 1] 0.05 micrometers in thickness It is 1.0 above. mum The manufacture approach of the Ti-aluminum system alloy sintered compact characterized by performing shaping processing, cleaning processing, and sintering processing in Ar ambient atmosphere after kneading Ti powder which performed the following nickel plating, and the mixed powder of aluminum powder with an organic binder.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the manufacture approach of a Ti-aluminum alloy sintered compact that use is expected as a lightweight heat-resisting material.
[0002]
[Description of the Prior Art] Efficient-izing and lightweight-ization are mentioned as a property required of a jet engine, an internal combustion engine, etc. Among these, about efficient-izing, it is required to raise an engine operating temperature. On the other hand, lightweight-ization cannot be attained although the current nickel radical heat-resistant alloy etc. is used. On the other hand, although the ceramics has come to be used for car motor from a viewpoint of lightweight-izing, dependability poses a problem from the point inferior to toughness. As an ingredient which solves these troubles, the intermetallic compound of a Ti-aluminum system has attracted attention in recent years. It is thought that a field of the invention will expand it from now on since this ingredient is lightweight, and excellent in high temperature strength and rich in toughness from the ceramics.
[0003] The manufacturing method of the sintered compact used as components poses a problem here. After making it the plate like a ferrous material, it was not able to be said that fabrication was carried out to the last configuration. Therefore, the so-called near net shape processing is needed. In this case, powder-metallurgy processing can be considered to be precision casting as the shaping approach (for example, "the reinforcement of an intermetallic compound, deformation", the Japan Institute of Metals symposium draft, 1988, P20).
[0004]
[Problem(s) to be Solved by the Invention] However, when using precision casting, since activity [Ti], a device is needed for an ambient atmosphere, refractories, etc. On the other hand, in general powder-metallurgy processing, manufacture of a complicated geometry component and large-sized components is difficult. Moreover, when performing a powder raw material by dissolution-ingot-grinding, a problem remains in the dissolution and an ingot manufacturing method as well as casting.
[0005] In recent years, the injection-molding method has attracted attention as the manufacture approach of the components of a complicated configuration metal or the ceramics. This is the approach of manufacturing a sintered compact through cleaning and a sintering process, after fabricating the raw material manufactured by carrying out heating kneading of a metal or ceramic fines, and the organic binder by the injection-molding method. However, utilization is not carried out in order to be anxious about penetration of C from an organic binder component, and degradation of the toughness by oxidation at cleaning and a sintering process about active metals, such as Ti. moreover -- although there is a report (a 1st super-environment-resistant advanced ingredient symposium draft (1990.10 months), P7) of injection molding which used Ti-aluminum alloy powder about injection molding of Ti-aluminum system alloy powder, even if the sintered compact of high density is obtained -- a front face -- there is a problem in description, dimensional accuracy, etc. Moreover, as the manufacture approach of Ti sintered compact by injection molding, the reaction of C and O is used for JP,2-54733,A by vacuum

sintering, and the method of reducing C and O is indicated. However, it is necessary to adjust C of a Plastic solid, and the amount of O by this approach. This invention aims at offering the manufacture approach of the Ti-aluminum alloy sintered compact which solved the above troubles.
[0006]
[Means for Solving the Problem] This invention is 0.05 micrometers in thickness. It is 1.0 above. mum After kneading Ti powder which performed the following nickel plating, and the mixed powder of aluminum powder with an organic binder, it is the manufacture approach of the Ti-aluminum system alloy sintered compact characterized by performing shaping processing, cleaning processing, and sintering processing in Ar ambient atmosphere.
[0007]
[Function] According to this invention, nickel which covered the powder front face prevents penetration of C in a kneading process, a forming cycle, and a cleaning process. The complicated configuration sintered compact of high density is obtained without degrading the quality of the material by this. The thickness of nickel plating is 0.05 micrometers. When it is the following, there is no above-mentioned effectiveness. Moreover, 1 mum If it exceeds, the above-mentioned effectiveness will be saturated and will degrade the quality of the material of sintering material. On the other hand, although especially the manufacture approach of Ti powder or aluminum powder that is a raw material is not specified, mean particle diameter is 30 micrometers. The following is desirable (refer to JP,2-54733,A). Since decomposition temperature etc. is not especially restricted, binding material may also use a well-known cementitious material. As the kneading approach, the high kneading machine of kneading force, such as a pressurized kneader and a continuous extrusion machine, is suitable. As the shaping approach, injection molding is effective about a small complicated geometry component. Moreover, when tabular components are required, extrusion molding and roll forming are suitable. At the time of sintering, since nickel reacts with Ti and reduces the melting point, it can make sintering temperature low.
[0008]
[Example] the inside of the electroless deposition liquid (PH5, 90 degrees C) which consists Ti powder with a mean particle diameter of 25 micrometers of chlorination nickel 30g/1, sodium hypophosphite 10g/1, and sodium citrates 10g/1 -- supplying -- predetermined time amount stirring -- it carried out. The acetone washed the powder after plating. In addition, the plating nickel component was 10% in weight %. This powder and aluminum powder with a mean particle diameter of 10 micrometers were kneaded with the pressurization mold kneader with thermoplastics, the wax, and the plasticizer after mixing by 65:35 at the weight ratio, and the injection-molding raw material was produced. After performing heating cleaning by carrying out injection molding of this to tabular [with die length of 80mm, a width of face / of 15mm /, and a thickness of 4mm], and carrying out a temperature up with the heating rate of 10 degrees C/h to 550 degrees C in nitrogen gas, sintering of 2 hours was performed at 1200 degrees C in Ar ambient atmosphere.
[0009] It was with Ti powder which does not perform nickel plating as comparison material, and the sintered compact was manufactured at this process (example 1 of a comparison). Moreover, the sintered compact was manufactured at this process using the powder which mixed the carbonyl nickel powder equivalent to the amount of plating with Ti powder and aluminum powder (example 2 of a comparison). Moreover, the case (example 3 of a comparison) where average plating thickness is 0.04 micrometers, and 1.2 micrometers (example 4 of a comparison) nickel-plating Ti powder were manufactured, and the sintered compact was manufactured at this process. From these sintering material, the test piece was processed and the mechanical property was investigated. Both quality-of-the-material property is shown as compared with Table 1. It turns out that the sintered compact by this invention is excellent in the reinforcement in an elevated temperature, and ductility compared with the example of a comparison.
[0010]
[Table 1]

	メッキ厚さ（μm）	相対焼結密度（%）	ＴＳ（室温）（kgf/mm² ）	Ｅｌ（室温）（%）	０．２%耐力（８００℃）（kgf/mm² ）
実施例１	０．１	９５	５８	１．８	４０
実施例２	０．５	９６	６２	１．５	４８
実施例３	０．８	９７	６４	１．２	５２
比較例１	０	９０	４０	０．２	３０
比較例２	０	９０	６０	０．１	４５
比較例３	０．０４	９０	４８	０．２	４０
比較例４	１．２	９０	６５	０．６	４２

[0011]
[Effect of the Invention] While manufacture of the lightweight heat-resisting-material components of a complicated configuration is attained by this invention and being able to measure engine high performance-ization, it leads to application expansion of an intermetallic compound.

[Translation done.]

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(54)【発明の名称】　Ｔｉ－Ａｌ系合金焼結体の製造方法

(57)【要約】
【目的】航空機のエンジンなどに用いるため、高温強度と靭性にすぐれたＴｉ－Ａｌ系金属間化合物による複雑形状の軽量耐熱材料部品の製造を可能とする。
【構成】金属粉末射出成形において、Ｎｉメッキを施したＴｉ粉末とＡｌ粉末とを混合後、有機バインダーと混練し、成形処理、脱脂処理、焼結処理を行なうことにより、Ｔｉ－Ａｌ系合金焼結体を得る。

【特許請求の範囲】

【請求項１】　厚さ0.05μm 以上1.0 μm 以下のＮｉメッキを施したＴｉ粉末と、Ａｌ粉末の混合粉末とを有機バインダーと混練したのち、成形処理、脱脂処理、Ａｒ雰囲気中での焼結処理を行なうことを特徴とするＴｉ－Ａｌ系合金焼結体の製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、軽量耐熱材料として利用が期待されているＴｉ－Ａｌ合金焼結体の製造方法に関する。

【０００２】

【従来の技術】ジェットエンジン、内燃機関等に要求される特性として高効率化、軽量化が挙げられる。このうち高効率化についてはエンジンの運転温度を高めることが必要である。これに対し、現在Ｎｉ基耐熱合金等が使用されているが、軽量化は達成できない。一方軽量化という観点から、自動車用エンジンにはセラミックスが使用されるようになってきたが、靭性に劣る点から信頼性が問題となる。これらの問題点を解決する材料として、近年Ｔｉ－Ａｌ系の金属間化合物が注目されてきた。この材料は軽量でかつ高温強度に優れ、セラミックスよりも靭性に富むため、今後利用分野が拡大していくと考えられる。

【０００３】ここで問題となるのが、部品となる焼結体の製造法である。鉄鋼材料のように板にしてから最終形状に成形加工するといったことは出来ない。従って、いわゆるニアネットシェイプ加工が必要となってくる。この場合、成形方法として精密鋳造と粉末冶金法が考えられる（例えば「金属間化合物の強度と変形」，日本金属学会シンポジウム予稿，１９８８，Ｐ２０）。

【０００４】

【発明が解決しようとする課題】しかしながら、精密鋳造法を利用する場合、Ｔｉが活性なため雰囲気、耐火物等に工夫が必要となる。一方、一般の粉末冶金法では複雑形状部品、大型部品の製造がむずかしい。また粉末原料を溶解ーインゴットー粉砕で行なう場合、鋳造法と同じく溶解およびインゴット製造法に問題が残る。

【０００５】近年、複雑形状金属やセラミックスの部品の製造方法として、射出成形法が注目されてきている。これは金属あるいはセラミックス微粉と有機バインダを加熱混練することにより製造した原料を射出成形法にて成形した後脱脂、焼結工程を経て焼結体を製造する方法である。しかしながらＴｉ等の活性金属については脱脂、焼結工程で有機バインダ成分からのＣの進入や、酸化による靭性の劣化が懸念されるため実用化はされていない。また、Ｔｉ－Ａｌ系合金粉末の射出成形については、Ｔｉ－Ａｌ合金粉末を用いた射出成形の報告（第１回超耐環境性先進材料シンポジウム予稿（１９９０・１０月），Ｐ７）があるが、高密度の焼結体は得られても表面性状、寸法精度などに問題がある。また、射出成形によるＴｉ焼結体の製造方法としては、特開平２－５４７３３号公報に真空焼結によりＣとＯの反応を用いて、Ｃ，Ｏを低減させる方法が開示されている。しかし、この方法では成形体のＣ，Ｏの量を調整する必要がある。本発明は、以上のような問題点を解決したＴｉ－Ａｌ合金焼結体の製造方法を提供することを目的とする。

【０００６】

【課題を解決するための手段】本発明は、厚さ0.05μm 以上1.0 μm 以下のＮｉメッキを施したＴｉ粉末と、Ａｌ粉末の混合粉末とを有機バインダーと混練したのち、成形処理、脱脂処理、Ａｒ雰囲気中での焼結処理を行なうことを特徴とするＴｉ－Ａｌ系合金焼結体の製造方法である。

【０００７】

【作用】本発明によれば、粉末表面を覆ったＮｉが混練工程、成形工程、脱脂工程でのＣの進入を防ぐ。これにより材質を劣化させることなく、高密度の複雑形状焼結体が得られる。Ｎｉメッキの厚さが0.05μm 未満の場合、上記効果がない。また1 μm を超えると、上記効果は飽和し、焼結材の材質を劣化させる。一方、原料であるＴｉ粉末やＡｌ粉末の製造方法は特に規定しないが、平均粒径は30μm 以下が望ましい（特開平２－５４７３３号参照）。結合材も特に分解温度等は制限されないため、公知のバインダー材を使用してよい。混練方法としては、加圧ニーダ、連続押出機等の混練力の高い混練機が適する。成形方法としては、小型複雑形状部品については、射出成形が有効である。また、板状の部品が必要な場合は押し出し成形、ロール成形が適する。焼結時ＮｉはＴｉと反応して融点を低下させるため、焼結温度を低くすることができる。

【０００８】

【実施例】平均粒径２５μｍのＴｉ粉末を塩化Ｎｉ３０ｇ／１、次亜リン酸ナトリウム１０ｇ／１、クエン酸ナトリウム１０ｇ／１からなる無電解メッキ液（ＰＨ５、９０℃）中に投入し、所定の時間攪拌した。メッキ後の粉末をアセトンで洗浄した。なお、メッキＮｉ成分は重量％にて１０％であった。この粉末と平均粒径１０μｍのＡｌ粉末を重量比にて６５：３５で混合後、熱可塑性樹脂，ワックス，可塑剤とともに加圧型ニーダにて混練し、射出成形原料を作製した。これを長さ８０ｍｍ，幅１５ｍｍ，厚さ４ｍｍの板状に射出成形し、窒素ガス中で５５０℃まで１０℃／ｈの加熱速度で昇温することにより加熱脱脂を行なった後、Ａｒ雰囲気中で１２００℃で２時間の焼結をおこなった。

【０００９】比較材としてＮｉメッキを施さないＴｉ粉末をもちいて同工程で焼結体を製造した（比較例１）。また、メッキ量に相当するカルボニルＮｉ粉末を、Ｔｉ粉末、Ａｌ粉末と混合した粉末を用いて同工程にて焼結体を製造した（比較例２）。また、平均メッキ厚さが

０．０４μｍの場合（比較例３）と１．２μｍ（比較例４）のニッケルメッキＴｉ粉末を製造し同工程にて焼結体を製造した。これらの焼結材より試験片を加工し、機械的特性を調査した。両者の材質特性を表１に比較して示す。本発明による焼結体は比較例に比べて高温での強度，延性に優れることがわかる。

【００１０】

【表１】

	メッキ厚さ（μｍ）	相対焼結密度（%）	ＴＳ（室温）(kgf/mm²)	Ｅｌ（室温）（%）	０．２%耐力（８００℃）(kgf/mm²)
実施例１	０．１	９５	５８	１．８	４０
実施例２	０．５	９６	６２	１．５	４８
実施例３	０．８	９７	６４	１．２	５２
比較例１	０	９０	４０	０．２	３０
比較例２	０	９０	６０	０．１	４５
比較例３	０．０４	９０	４８	０．２	４０
比較例４	１．２	９０	６５	０．６	４２

【００１１】

【発明の効果】本発明により複雑形状の軽量耐熱材料部品の製造が可能となり、エンジン等の高性能化が計れるとともに、金属間化合物の用途拡大につながる。